

APPLICATION

FOR

UNITED STATES LETTERS PATENT

FOR

PROCESS AND APPARATUS FOR PRODUCTION OF SILICA GRAIN
HAVING DESIRED PROPERTIES AND THEIR FIBER OPTIC
AND SEMICONDUCTOR APPLICATIONS

BY

KIRIL A. PANDELISEV

James C. Wray, Reg. No. 22,693
Meera P. Narasimhan, Reg. No. 40,252
1493 Chain Bridge Road
Suite 300
McLean, Virginia 22101
Tel: (703) 442-4800
Fax: (703) 448-7397

09831092-061501

Process and Apparatus for Production of Silica Grain
Having Desired Properties and Their Fiber Optic
and Semiconductor Applications

BACKGROUND OF THE INVENTION

This application claims the benefit of U.S. Provisional Application No. 60/258,494, filed December 29, 2000.

Silica powders are used in the production of silica glass products such as optical windows, scintillators and optical filters.

Fine control of the processes is required to produce end products with desired characteristics. Variations in characteristics result in products with little or no economic value.

Needs exist for better starter materials to ensure uniformity of products.

SUMMARY OF THE INVENTION

Silica grains of desired sizes and desired compositions and doping for use as starter materials in silica products are produced using the present invention.

According to the invention, silica powders are introduced or created in a vacuum chamber.

A gas or gases plasma heats the powders rendering them sticky. The surfaces melt and the powder particles agglomerate and fuse into larger particles as they pass through the plasma.

Microwave/electron cyclotron resonance (MW-ECR) or other methods for generating plasma may be introduced in the chamber. Argon, nitrogen, ammonia, oxygen and other gasses may be used for the plasma. One or more sources producing the same or different gas plasma may be coupled with the same chamber. The plasma ports all may be directed at the same chamber section, or they may be cascaded in specific orders. Each chamber may contain one or

more plasma generators resulting in certain plasma density. Such chambers may form a cascade. Fused silica grains traveling through the cascade may experience increase or decrease in temperature. The same vacuum or different vacuums may be present at each plasma port. The plasma ports may be within one chamber or they may be in separate chambers. Chambers may be separated or not separated by gate valves. Plasma chamber cascades may be employed to achieve the desired grain properties. The plasma flow may consist of pure plasma, plasma and carrier gas, or plasma and neutral gas. The plasma may have, plasma and any mixture of neutral gases. The plasma density and temperature may be adjusted to fit the grain size of the fused silica introduced in the chamber in order to obtain certain desired grain size, grain size distribution and OH content.

Microwave electron cyclotron resonance plasma (MW-ECR) sources, among any other plasma generators may be used for production of synthetic fused silica grain of desired size or for processing of natural quartz powder into powder with certain grain size and quality. The plasma source used will allow for clean, temperature and density controlled stream of plasma that will allow for controlling the fused silica or natural quartz grain temperature for certain periods of time.

Synthetic fused silica powder may be introduced in the chamber as powder, powder and plasma mix, powder obtained via pyrolysis of silicon tetrachloride, silicon tetra fluoride, organosilicate compounds, and other silicon based compounds, organic or inorganic. When subjected to heat, plasma stream, EM field or other methods suitable for this purpose the powder will result in fused silica particles having the desired purity, OH content and particle size distribution.

Ion temperatures in the vicinity of 1 eV and electron temperatures between 4-7 eV may be used. The density of the plasma ($\sim 10^{10} \text{ cm}^{-3}$) and its temperature are determined by the plasma source array and the placement of each plasma generator and they will determine the temperature of the silica grain and how much it will fuse into larger grains of silica. A plasma exposure

cascade may further enhance the grain size to the desired grain volume or grain weight. Heating individual grains to such high temperatures before the fusion and after the fusion and possibly repeating this process within a cascade of plasma exposure eliminates OH group presence in the fused silica and the reaction with various gases in plasma or neutral state can further purify the silica grain and the soot produced from the same. Repeat of the silica grain with plasma/neutral gas interaction, and the appropriate time length for the contact will determine the appropriate temperature of the reaction taking place and the fusion between different grains into grains having desired grain size and purity. Reactive plasma such as atomic chlorine, fluorine and other ions may be used to remove certain impurities in the fused silica grain.

Dopant may be introduced in gaseous, liquid or solid state for doping of the grains while they are fusing or as a later step in the fused silica processing.

Additional grain heating by means of resistive, RF or any other heating methods may be used. Multi zone heating arrangement in the chamber may be applied for this purpose. Equilibrium chamber vacuum or differential vacuum may be present during the synthetic fused silica or natural quartz grain processing.

The so purified material can remain in granular state, or it can be deposited on a bait that can be made of quartz, graphite, silicon carbide, ceramic, metal or metal alloy that possesses any porosity: from very porous to solid material. The bait can have any desired shape and cross section to better suit the step processing of the fused silica.

In one embodiment synthetic fused silica or natural quartz powder may be introduced simultaneously. The plasma heated powder jets from plurality of ports enter the chamber and they are "contained" within certain elliptically shaped cloud. Other plasma for additional grain heating and purification may be introduced in the particle cloud. Reactive gasses in plasma or neutral state may also be introduced in the particle cloud for purification or other purposes.

The grains collide among themselves forming larger grains. The high temperature of the grain provides for removal of the OH content in the grain. The high temperature of the grain and

the reactions the grain is subjected to in the particle cloud or in the chamber in general provides for removal of various trace elements that are pumped out in gaseous form.

Such produced grain may be subjected to a cascade of individual or interconnected chambers that further contribute to the grain size, grain size distribution and grain purity.

In another embodiment the powder is deposited in a tray that can be heated. Synthetic fused silica or natural quartz having desired purity, OH content and grain size distribution is obtained. This powder can further be used in various processes for fabrication of fiber optic preforms, synthetic fused silica, natural quartz or their combination made into tubes for modified chemical vapor deposition (MCVD), for fabrication of fiber optic preforms, doped or undoped cores for axial vapor deposition methods, for fiber optic preforms fabrication, solid rods and plate shaped members for semiconductor wafers and optical components fabrication.

These and further and other objects and features of the invention are apparent in the disclosure, which includes the above and ongoing written specification, with the claims and the drawings.

BRIEF DESCRIPTION OF THE DRAWINGS

Figures 1-16 discuss different embodiments for the synthetic fused silica fabrication, applications and products made by the same.

Figure 1 show forming of fused silica grains from powder particles.

Figure 2 shows collecting, treating and processing the fused silica grains.

Figure 3 adds electrodes and an electric field to the softened fused silica.

Figure 4 shows double crucible used in the process.

Figure 5 shows direct plate or bar formation.

Figure 6 is a schematic perspective representation of a porous preform-general chamber, which may be horizontal, vertical or any other position.

Figure 7 shows a cross-sectional view of the chamber shown in Figure 1, in which one or

a plurality of deposition rods made from carbon, SiC, ceramic or graphite may be rotated to collect the glass soot.

Figure 8 shows spacing of plural preforms in a chamber.

Figure 9 shows multiple preforms with rotation and translation in the silica grain streams in the chamber.

Figure 10 shows dopant gas distribution to and through the preform.

Figure 11 shows rotating and translating the preform of in powder streams and forming a cladding layer.

Figure 12 shows vitrifying and densifying a cladding layer on a core.

Figures 13A-13D show transforming a tubing into a solid member.

Figures 14A and 14B show transforming a tubing into a solid member.

Figures 15A and 15B show vitrifying a silica tube and the product produced.

Figure 16 schematically shows forming a plate or bar from a tubular or rod preform formed from fused silica grains.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

Figure 1 shows a chamber 300 with burners 3 and small grain silica powder introduction ports 37. The burners 3 create fine silica powder from precursor materials and heat the plasma 311. A differential reduced pressure 301 is drawn on the chamber using valved vacuum line 303. A valved gas inlet 305 provides dopant gas and inert gas. A valved vent 307 removes combustion gasses and excess dopant gas. Microwave electron cyclotron resonance heaters 309 create the high temperature plasma 311. The fine grain silica powders pass through the plasma and are heated and softened. The hot soft surfaces of the fine grain powder particles cause agglomeration and fusing of the powder particles into large grain silica particles. Uniform grain size is created, and OH content is reduced or eliminated. The plasma fields are controlled so that surface melting of the increasing size particles is maintained in the plasma. The plasma 311

contains multiple heat zones. Multizone resistance or radio frequency (RF) heaters 309 may be used to maintain temperatures in plasma fields 311. The fused particles are collected in a heated rotating tray 313 which is rotated clockwise or counter clockwise or in alternating directions and elevated and lowered as shown by arrows with a turning and elevating device 314.

The first chamber produces silica and other soot of desired size. The vacuum chamber has plurality of vacuum ports, gas inlet ports, vent ports, reactive burners, and silica powder delivery ports. The chamber is heated by resistance or RF heating, plasma heating or any other mean of heating, connected through plurality of feedthroughs. Crucible made from graphite, silicon carbide, ceramic material, metal or metal alloys receives the material. The vacuum chamber can be multiple chambers.

Figure 2 shows a chamber 183 for producing silica powder 185 and other metal oxides from burners 3 and from soot 187 introduced from ports and agglomerated in plasma 189 into grains having desired particle size. Fine oxide particles, in suit made from burners 3 or delivered through plurality of ports 37 on the chamber are heated in plasma 189 and allowed to recombine.

The plasma 189 is created by hot temperatures produced in inert gases by heating in a multizone arrangement. Depending on the time the particles stay hot and the distance the particles travel, they recombine into larger grains of desired size. The vacuum chamber 183 has multizone heating zones Z1-Z6 with heaters 184. Microwave electron cyclotron resonance heating, in zones Z1, Z2 and Z3 of increasing temperatures, resistive heating, RF heating of the plasma 189 or other heating methods of the grains may be employed.

The soot is collected in a crucible 191. A heater 193 in zone Z4 keeps the sized grains hot in crucible 191. The hot grains are doped using a dopant injector 195, as shown in Figure 2. The grains 185 may be melted 196, funneled and flowed around a former 197 and filled with an inert gas with a dopant 199 or an inert gas 199 to form a tube 201. Tube 201 passes out of chamber 183 through a gate 202 after solidification in zone Z6 in which temperatures are maintained by heaters 198.

09881092 061501
105190 26018860

The vacuum chamber having plurality of vacuum ports, gas inlet ports, vent ports, reactive burners, and silica powder delivery ports. The chamber is heated by resistance heating, RF heating, plasma heating or any other means of heating connected through plurality of feedthroughs. A funnel made from graphite, silicon carbide, ceramic material, metal or metal alloys receives the material. The material is softened in the funnel and transformed into a fused quartz article of choice. The fused silica article can rotate clockwise or counterclockwise. This material can feed into a fabrication apparatus.

Another chamber employing the new soot grain enlargement process for tube or rod fabrication is as shown in Figure 3. In that embodiment electric field generator 177 with electrodes 179 and 181 provides an electric field across the softened fused silica flow 125. Electrode 179 is located within the softened bubble 125 which forms tube 201. Electrode 181 is located outside the bubble 125. A plasma tube surface removal unit 204 cleans the surface of the tube in a hot plasma.

Figure 4 shows a double crucible 203 in the chamber. A vacuum chamber 183 having plurality of vacuum ports, gas inlet ports, vent ports, and a fused silica feed material introduction port is heated by resistance or RF heating or any other means of heating, connected through plurality of feedthroughs. A second crucible 203 made from graphite, silicon carbide, ceramic material, metal or metal alloys receives, holds and melts the material from the feed crucible 191, softens the same and remelts the material. A dopant gas from tube 195 is added to the molten material in crucible 203. A fused silica tube is produced. Pluralities of ultrasound generators 206 are in contact with the crucible to provide proper mixing and outgassing. Additional vacuum ports are placed above the softened material to remove any gas bubbles. The chamber can be a single chamber or plurality of chambers.

Figure 5 shows a plate or bar forming chamber 211 in which the infeed is a tube 201 or rod. The plate or bar forming chamber 211 directly coupled to chamber 183 for receiving the fused silica tube input 217 directly from the output of chamber 183.

The plate/bar fabrication chamber 211 has two separated chambers. A vacuum chamber 213 having plurality of valved vacuum ports 221, gas inlet ports 223, vent ports 225 and a fused silica feed material 217 introduction port 227 is heated by resistance of RF heating 219 or any other means of heating, connected through a plurality of feedthroughs. A crucible 230 made from graphite, silicon carbide, ceramic material, metal or metal alloys receives the material 231 from the feed tube 217, softens, dopes, degasifies and solidifies the material. A fused silica plate or a bar 210 is produced. A plurality of ultrasound generators 233 are in contact with the crucible to promote proper mixing and outgassing. Additional vacuum ports 235 are placed above the softened material to remove any gas bubbles. The chamber can be a single chamber or plurality of chambers 213, 215 with sequentially controlled heat zones.

The plate/bar fabrication chamber is a vacuum chamber having plurality of vacuum ports, gas inlet ports, and vent ports. A fused silica feed material introduction port is heated by microwave, resistance, RF heating, or any other means of heating, connected through plurality of feedthroughs. A crucible made from graphite, silicon carbide, ceramic material, metal or metal alloys receives the material from the feed rod, softens the same and solidifies the material. A fused silica plate or a bar is produced. Plurality of ultrasound generators are in contact with the crucible to promote proper mixing and outgassing. Additional vacuum ports are placed above the softened material to remove any gas bubbles. The chamber can be a single chamber or plurality of chambers.

Figures 6 and 7 show a plurality of substrates 11 with controlled temperature housed in a vacuum chamber 1. A plurality of burners 3 for oxidation 5 of metal halides 7 such as SiCl_4 , SiF_4 and others are either imbedded in the chamber wall 8 or they are placed inside the chamber. The proximity of the burners to the substrates 11 as well as the distance of the substrates from the center 9 of the chamber are optimized based on the number of the substrates 11, the number of the burners 3 and their relative positions. The chamber 1 may have round, rectangular or any other suitable shape that is needed to optimize the process. Vacuum ports 13 with valves 15,

vents 17 with valves 19 and a plurality of gas inlet ports 21 with valves 23 are also added to the chamber. The chamber may be vertical, horizontal, sloped and any other position or combination suitable for the new process. The chamber walls 8 may have a cooling jacket 25 for temperature control and appropriate venting apparatus for the gasses generated during the deposition. Appropriate openings are provided at one end, at each end or on one or two sides of the chamber for loading and unloading of the chamber.

A plurality of power feeds for resistive heating 29 or RF coils 31 and appropriate power feedthroughs 33 and shields 35 are also included in the chamber.

The chamber may have plurality of ports 37 for introduction of soot 39 made during another operation.

The chamber and the substrate assembly may be rotated in respect to each other clockwise or counterclockwise at certain desired speeds. Each substrate may be rotated around its axis clockwise or counterclockwise at certain desired speeds. All rotations are aimed at establishing conditions for good thickness and uniformity properties of the deposited material in the porous perform 41.

Figure 9 shows a tubular substrate 11 with deposited material 43. Each substrate 11 may be made of solid, porous or perforated material made from graphite, silicon carbide, ceramic, metal or metal alloys. It may have round, rectangular or any other cross section. It may be tubular, solid or tubular with solid core made from the same or other material. The ends 45 may have the same cross section throughout, or the ends may have different dimensions or shapes. The ends 45 may be mechanically connected to the substrate 11 or they may be part of the substrate. A gas line 47 or vacuum line may be connected with the hollow portion of each substrate having tubular shape, with or without a central rod.

Figure 10 shows an apparatus consisting of a vacuum chamber 51 having plurality of vacuum ports 53, vent lines 55, and gas ports 57 doping ports 59 for purging and doping purposes, plurality of power feedthroughs 61 with or without cooling lines 63 in them for

resistive, RF 65 or any other form of heating the substrate 11 of the preform 41 and the preform itself. The chamber may have multiple heating zones 67 to accommodate the process being performed there. Rotation and translation mechanisms 60 rotate 62 and translate 64 the substrate 11 and preform 41. Slip rings 66 conduct power from source 68 to heat the substrate 11.

In Figure 10 the dopant gases 58 surround the preform 41, and purge or dopant gases 56 from purge or dopant line 54 flow outward from the porous substrate through the porous preform 41.

As shown in Figure 11, a doped or undoped cladding layer 77 may be added to a doped or undoped preform core silica deposit 75. Several preforms 41 may be constructed at the same time using the independent rotation mechanism and support 70.

As shown in Figure 12, the core-forming silica layer 75 may be vitrified 76 initially before deposition of the cladding layer 77, followed by vitrification 78 of the cladding layer, all within the single chamber 51. The independent rotation mechanism 70 permits deposit and vitrification of layers on multiple preforms concurrently.

Figures 13A and 13B show cross-sections of tube-shaped preforms 41 with a hole 81, an inner tubular layer 83, and an outer tubular layer 85. Supporting the preform 41 between ends, heating the preform to softening temperature and rotating the preform shrinks the preform to the solid member 86 with a solid core 87 and cladding 89, as shown in Figures 13C and 13D.

Figure 15A shows a vitrified silica tube 90 in a chamber 51. The vitrified tube 90 is removed from the chamber, as shown in Figure 15B. Detaching the independent rotation mechanism from support ends 45 allows the substrates to be detached from the mechanism 70. Alternatively, the mechanism may be left in place on the support 45 while the individual substrates 11 are removed.

When the substrate is fused silica, the tube is ready to be used or ready to be softened and to be compacted and densified into a solid.

Alternatively, the substrate 11 may be heated, and the fused silica tube 90 may be slid off

the substrate after a film is melted adjacent the substrate, after the ends 91 are removed as shown in Figures 12A and 12B.

The tubing 90 that is removed has a hole 93 and a tube wall 95, as shown in Figure 13A, before it is compressed into a solid doped fused silica rod 97, as shown in Figure 13B.

Figures 14A and 14B show fusing a doped fused silica tubing 90 to a doped fused silica rod 97.

Figure 16 shows a plate/bar fabrication chamber 211. A vacuum chamber 213 having plurality of valved vacuum ports 221, gas inlet ports 223, vent ports 225 and a fused silica feed material 217 from introduction port 227 is heated by resistance or RF heating 219 or any other means of heating, connected through a plurality of feedthroughs. A crucible 230 made from graphite, silicon carbide, ceramic material, metal or metal alloys receives the material 231 from the feed tube 217, and softens, dopes, degasifies and solidifies the material. A fused silica plate or a bar 210 is produced. A plurality of ultrasound generators 233 are in contact with the crucible to prevent proper mixing and outgassing. Additional vacuum ports 235 are placed above the softened material to remove any gas bubbles. The chamber can be a single chamber or plurality of chambers 213, 215 with sequentially controlled heat zones.

Figure 16 also shows a plate or bar forming chamber 211 in which the infeed 217 is a solid rod.

The heating of the substrate may be accomplished by separate heaters positioned axially along or in the substrate. Alternatively if resistance heating is used, the heating wire may be varied in shape, form or size along the length of the substrate. The substrate may be linear or planar and may be made in one element or plural elements. A single control or multiple independent controls may be used. The varied heating of the substrate may be used to effect

uniformity of the preform in an axial direction. Alternatively the varied heating may be used to effect varied densities or porosities of the perform along it's length or per unit area.

EXAMPLES

Silica Glass Body Fabrication

Production of synthetic fused silica glass bodies having controlled density and desired size and shape have been of interest to the natural quartz or synthetic fused silica glass industry for some time. The densities of the formed silica body mainly depend on the temperature of the flame, the distance between the substrate and the burner, and rotational and translational speeds of the substrate. Densities between 10% and 30% have been reported by this approach. The size of the body and the optimal ratio between the wall thickness (W_t) and the outside diameter (D_o), W_t/D_o , as well as the ratio between the outside diameter (D_o) and the Inside diameter (D_i), D_o/D_i , and the way the body is held during the deposition depend greatly on the density of the body surface temperature and the body density.

To overcome the current limitations and to produce large glass bodies made from synthetic fused silica, natural quartz or combination thereof, substrate heating and surface heating has been introduced. The amount of the surface heating will greatly depend on the substrate temperature, the chamber pressure, the size of the quartz particles and their temperature at impact of the surface and the size of the quartz member fabricated. Silica preforms, doped or undoped, having desired density and optimized diameter ratio can be fabricated following the examples shown below.

Example No. 1: Silica Body Fabrication

A heated substrate having temperature of about 1000°- 1400 °C is subjected to plurality of silica particle stream either generated in situ by high temperature reactions of silica precursors, or fabricated in a separate process and then introduced via ports on the chamber in pure form, doped form, mixed with neutral gas, gas plasma or combination thereof. The so accelerated particles collide with the substrate and deposit themselves on the substrate. Subsequent particles deposit on the material already deposited, and layer by layer the silica member is formed. The silica particle stream may be doped or undoped. The temperature of the substrate might be sufficient to keep the surface of the so formed body at the same temperature. The silica body so formed is hot enough to allow for formation of a solid fused silica body. Densities between 80% and 100% may be expected as a result.

The substrate may be tubular or solid form having the desired diameter and cross section. Desired ratios between the outside and inside diameters may be obtained using this method. If tubular, the substrate may be solid or porous, depending on the dopant or reactive gas flow desired. This achieves optimized silica material-to-gas contact. The hot substrate may also serve as a heater for the dopant gas and increased reaction time. Porous substrates can also diminish the possibility of gas bubbles entrapment near the surface of the substrate.

Substrate and surface temperatures between about 700°C and 1600°C may result in various silica densities from 10% to 100%. Controlling the fused silica body temperature by controlling the substrate and surface temperature may result in control of the pore size and pore

density in the material. If the variation is in the radial direction, exposure to dopant gas over periods of time will result in radial gradient of the dopant distribution. By doing so silica members having radially graded indexes of refraction may be fabricated.

If the substrate is other than a silica core, doped or undoped made from fused silica or natural quartz; the resulting silica member may be in tubular form or may be in solid form after collapsing the tube.

Employing non uniform substrate heating along the length of the body, one may obtain a silica member having variable density over its length.

Example No. 2: Doped and Undoped Layer Combination Silica Body

Fabrication

Step 1. Rotating and translating, a substrate consisting of porous tubing is heated to a temperature of about 1300 °C and is subjected to plurality of silica particle stream introduced via ports on the chamber. The accelerated particles collide with the substrate and deposit themselves on the substrate. Subsequent particles deposit on the material already deposited, and layer by layer the silica member is formed. A porous silica body having about 25-35% solid glass density is obtained by this process.

Step 2. Introducing silicon tetra fluoride, SiF₄, through the porous substrate and/or the chamber into the deposited porous silica material for about 0.3 to 6 hours at temperature of about 800-1400 °C, the silica material is doped.

Step 3. The substrate and/or chamber temperature is raised to about 1400-1600 °C while

rotating the substrate. A vitrified tubular silica body having desired wall thickness is formed.

Step 4. The so formed vitrified tubular silica body is heated to temperature of about 1300 °C and is subjected to plurality of silica particle streams introduced via ports on the chamber. The accelerated particles collide with the substrate and deposit themselves on the substrate. Subsequent particles deposit on the material already deposited and layer by layer the silica member is formed. A porous silica body having about 25-35% solid glass density is obtained by this process.

Step 5. The substrate and/or chamber temperature is raised to about 1400-1600 °C while rotating the substrate. The newly deposited porous silica is vitrified, and a tubular silica body having desired doped inner wall thickness IW_t and undoped other wall OW_t desired wall thickness is formed. The duration of the silica deposition for certain substrate cross sections and sizes can be adjusted to allow for various ratios between the wall thicknesses of the doped and undoped portion of the tubular member, e.g., 1:2, 1:3, 1:5, etc.

Example No. 3: Doped non-porous and undoped porous layer combination silica body fabrication

Step 1. Rotating and translating, a substrate consisting of porous tubing is heated to a temperature of about 1300 °C and is subjected to plurality of silica particle stream introduced via ports on the chamber. The accelerated particles collide with the substrate and deposit themselves on the substrate. Subsequent particles deposit on the material already deposited, and layer by layer the silica member is formed. A porous silica body having about 25-35% solid glass density is obtained by this process.

Step 2. Introducing silicon tetra fluoride, SiF_4 , through the porous substrate and/or the chamber into the deposited porous silica material for about 0.3-6 hours at temperature of about 800-1400 °C, the silica material is doped.

Step 3. The substrate and/or chamber temperature is raised to about 1400-1600 °C while rotating the substrate. A vitrified tubular silica body having desired wall thickness is formed.

Step 4. The so formed vitrified tubular silica body is heated to temperature of about 1300 °C and is subjected to plurality of silica particle streams introduced via ports on the chamber. The accelerated particles collide with the substrate and deposit themselves on the substrate.

Subsequent particles deposit on the material already deposited and layer by layer the silica member is formed. A porous silica body having about 25-35% solid glass density is obtained by this process. The duration of the silica deposition for certain substrate cross sections and sizes can be adjusted to allow for various ratios between the wall thicknesses of the doped and undoped portion of the tubular member, e.g., 1:2, 1:3, 1:5, etc.

Example No. 4: Undoped core and Fluorine doped cladding fiber optic preform fabrication

Step 1. Rotating and translating, a substrate consisting of porous tubing is heated to a temperature of about 1300 °C and is subjected to plurality of silica particle stream introduced via ports on the chamber. The accelerated particles collide with the substrate and deposit themselves on the substrate. Subsequent particles deposit on the material already deposited and layer by layer the silica member is formed. A porous silica body having about 25-35% solid glass density is

obtained by this process.

Step 2. The substrate and/or chamber temperature is raised to about 1400-1600 °C while rotating the substrate and maintained there for certain time interval. A vitrified tubular silica body having desired wall thickness is formed.

Step 3. The so formed vitrified tubular silica body is heated to temperature of about 1300 °C and is subjected to plurality of silica particle streams introduced via ports on the chamber. The accelerated particles collide with the substrate and deposit themselves on the substrate.

Subsequent particles deposit on the material already deposited and layer by layer the silica member is formed. A porous silica body having about 25-35% solid glass density is obtained by this process.

Step 4. Introducing silicon tetra fluoride, SiF_4 , through the porous substrate and/or the chamber into the deposited porous silica material for about 0.3-6 hours at temperature of about 800-1400 °C, the silica material is doped.

Step 5. The substrate and/or chamber temperature is raised to about 1400-1600 °C while rotating the substrate. The newly deposited porous silica is vitrified, and a tubular silica body having desired doped inner wall thickness IW_t and undoped outer wall OW_t desired wall thickness is formed.

Step 6. The substrate is transferred out of the deposition chamber area, and the substrate is removed. If wetting between the substrate and silica occurs, the substrate is heated to the softening point of the silica. The contact between the substrate and the silica member is melted and the substrate is removed.

Step 7. The so formed silica member is collapsed and a solid rod like silica member is

09881092-061501
TOST90-26T8860

formed. Undoped core (high index of refraction material) surrounded by fluorine doped cladding (low index of refraction material) having desired diameter and length is formed. The duration of the silica deposition for certain substrate cross sections and sizes can be adjusted to allow for various ratios between the core diameter and the outside cladding layer diameter of the fiber optic preform, e.g., 1:2, 1:3, 1:5, etc. The length of the chamber and the translation capabilities can provide basis for fabrication fiber optic preforms that are up 6 inches or more in diameter and several meters in length.

Example No. 5: Doped core and Fluorine doped cladding fiber optic preform fabrication

Step 1. Rotating and translating, a substrate consisting of porous tubing is heated to a temperature of about 1300 °C and is subjected to plurality of silica and dopant particle stream introduced via ports on the chamber. The accelerated particles collide with the substrate and deposit themselves on the substrate. Subsequent particles deposit on the material already deposited and layer by layer the silica member is formed. A porous silica body having about 25-35% solid glass density is obtained by this process.

Step 2. The substrate and/or chamber temperature is raised to about 1400-1600 °C while rotating the substrate and maintained there for certain time interval. A vitrified tubular silica body having desired wall thickness is formed.

Step 3. The so formed vitrified tubular silica body is heated to temperature of about 1300 °C and is subjected to plurality of silica particle streams introduced via ports on the chamber. The

accelerated particles collide with the substrate and deposit themselves on the substrate.

Subsequent particles deposit on the material already deposited and layer by layer the silica member is formed. A porous silica body having about 25-35% solid glass density is obtained by this process.

Step 4. Introducing silicon tetra fluoride, SiF_4 , through the porous substrate and/or the chamber into the deposited porous silica material for about 0.3-6 hours at temperature of about 800-1400 °C, the silica material is doped.

Step 5. The substrate and/or chamber temperature is raised to about 1400-1600 °C while rotating the substrate. The newly deposited porous silica is vitrified, and a tubular silica body having desired doped inner wall thickness IW_t and undoped outer wall OW_t desired wall thickness is formed.

Step 6. The substrate is transferred out of the deposition chamber area and the substrate is removed. If wetting between the substrate and silica occurs, the substrate is heated to the softening point of the silica. The contact between the substrate and the silica member is melted, and the substrate is removed.

Step 7. The so formed silica member is collapsed and a solid rod like silica member is formed. Undoped core (high index of refraction material) surrounded by fluorine doped cladding (low index of refraction material) having desired diameter and length is formed. The duration of the silica deposition for certain substrate cross section and size can be adjusted to allow for various ratios between the core diameter and the outside cladding layer diameter of the fiber optic preform, e.g., 1:2, 1:3, 1:5, etc. The length of the chamber and the translation capabilities can provide basis for fabrication fiber optic preforms that are up 6 inches or more in diameter and

several meters in length.

Example No. 6: Doped core and Fluorine doped graded index of refraction cladding fiber optic preform fabrication

Step 1. Rotating and translating, a substrate consisting of porous tubing is heated to a temperature of about 1300 °C and is subjected to plurality of silica and dopant particle stream introduced via ports on the chamber. The accelerated particles collide with the substrate and deposit themselves on the substrate. Subsequent particles deposit on the material already deposited and layer by layer the silica member is formed. A porous silica body having about 25-35% solid glass density is obtained by this process.

Step 2. The substrate and/or chamber temperature is raised to about 1400-1600 °C while rotating the substrate and maintained there for certain time interval. A vitrified tubular silica body having desired wall thickness is formed.

Step 3. The so formed vitrified tubular silica body is heated to temperature of about 1300 °C and is subjected to plurality of silica particle stream introduced via ports on the chamber. The accelerated particles collide with the substrate and deposit themselves on the substrate. Subsequent particles deposit on the material already deposited and layer by layer the silica member is formed. A porous silica body having about 25-35% solid glass density is obtained by this process.

Step 4. Introducing silicon tetra fluoride, SiF_4 , through the porous substrate and/or the chamber into the deposited porous silica material for T_1 hours at temperature of 800-1400 °C, the

silica material is doped. T_1 is about 0.3 to 2 hours.

Step 5. The substrate and/or chamber temperature is raised to about 1400-1500 °C while rotating the substrate. The newly deposited porous silica is vitrified, and a tubular silica body having desired doped inner wall thickness IW_t and undoped outer wall OW_t desired wall thickness is formed.

Step 6. The so formed vitrified tubular silica body is heated to temperature of about 1300 °C and is subjected to plurality of silica particle streams introduced via ports on the chamber. The accelerated particles collide with the substrate and deposit themselves on the substrate. Subsequent particles deposit on the material already deposited and layer by layer the silica member is formed. A porous silica body having about 25-35% solid glass density is obtained by this process.

Step 7. Introducing silicon tetra fluoride, SiF_4 , through the porous substrate and/or the chamber into the deposited porous silica material for $T_2 > T_1$ hours at a temperature of about 1100°C-1400 °C, the silica material is doped. T_2 is about 0.4 – 4 hours.

Step 8. The substrate and/or chamber temperature is raised to about 1400-1600 °C while rotating the substrate. The newly deposited porous silica is vitrified, and a tubular silica body having desired doped inner wall thickness IW_t and undoped outer wall OW_t desired wall thickness is formed.

Step 9. The so formed vitrified tubular silica body is heated to temperature of about 1300 °C and is subjected to plurality of silica particle streams introduced via ports on the chamber. The accelerated particles collide with the substrate and deposit themselves on the substrate. Subsequent particles deposit on the material already deposited and layer by layer the silica

member is formed. A porous silica body having about 25-35% solid glass density is obtained by this process.

Step 10. Introducing silicon tetra fluoride, SiF_4 , through the porous substrate and/or the chamber into the deposited porous silica material for $T_3 > T_2$ hours at temperature of about $1100^\circ\text{C} - 1400^\circ\text{C}$, the silica material is doped. T_3 is about 0.5 – 5 hours.

Step 11. The substrate and/or chamber temperature is raised to about $1400-1600^\circ\text{C}$ while rotating the substrate. The newly deposited porous silica is vitrified, and a tubular silica body having desired doped inner wall thickness IW_t and undoped outer wall OW_t desired wall thickness is formed.

Step 12. The so formed vitrified tubular silica body is heated to temperature of about 1300°C and is subjected to plurality of silica particle streams introduced via ports on the chamber. The accelerated particles collide with the substrate and deposit themselves on the substrate. Subsequent particles deposit on the material already deposited and layer by layer the silica member is formed. A porous silica body having about 25-35% solid glass density is obtained by this process.

Step 13. Introducing silicon tetra fluoride, SiF_4 , through the porous substrate and/or the chamber into the deposited porous silica material for $T_4 > T_3$ hours at temperature of about $1100^\circ\text{C} - 1400^\circ\text{C}$, the silica material is doped. T_4 is about 0.6 to 6 hours

Step 14. The substrate and/or chamber temperature is raised to $1400-1600^\circ\text{C}$ while rotating the substrate. The newly deposited porous silica is vitrified and a tubular silica body having desired doped inner wall thickness IW_t and undoped outer wall OW_t desired wall thickness is formed.

00001092-051504
T05T90-260T8860

Steps 15-17. Repeat Steps 12-14 while further reducing the exposure to gaseous dopant, SiF₄ in this case.

Step 18. The substrate is transferred out of the deposition chamber area and the substrate is removed. If wetting between the substrate and silica occurs, the substrate is heated to the softening point of the silica. The contact between the substrate and the silica member is melted and the substrate is removed.

Step 19. The so formed silica member is collapsed and a solid rod like silica member is formed. Undoped core (high index of refraction material) surrounded by graded index of refraction fluorine doped cladding (low index of refraction material) having desired diameter and length is formed. The duration of the silica deposition for certain substrate cross section and size can be adjusted to allow for various ratios between the core diameter and the outside cladding layer diameter of the fiber optic preform, e.g., 1:2, 1:3, 1:5, etc. The length of the chamber and the translation capabilities can provide basis for fabrication fiber optic preforms that are up to 6 inches or more in diameter and several meters in length.

Example No. 7: Doped core having graded index of refraction and Fluorine doped graded index of refraction cladding fiber optic preform fabrication

Step 1. Rotating and translating, a substrate consisting of porous tubing is heated to a temperature of about 1300 °C and is subjected to plurality of silica and dopant particle streams introduced via ports on the chamber. The accelerated particles collide with the substrate and deposit themselves on the substrate. Subsequent particles deposit on the material already

deposited and layer by layer the silica member is formed. A porous silica body having about 25-35% solid glass density is obtained by this process.

Step 2. The substrate and/or chamber temperature is raised to about 1400-1600 °C while rotating the substrate and maintained there for certain time interval. A vitrified tubular silica body having desired wall thickness is formed.

Step 3. Rotating and translating, a substrate consisting of porous tubing is heated to a temperature of about 1300 °C and is subjected to plurality of silica particle streams and reduced dopant particle streams introduced via ports on the chamber. The accelerated particles collide with the substrate and deposit themselves on the substrate. Subsequent particles deposit on the material already deposited and layer by layer the silica member is formed. A porous silica body having about 25-35% solid glass density is obtained by this process.

Step 4. The substrate and/or chamber temperature is raised to about 1400-1600 °C while rotating the substrate and maintained there for certain time interval. A vitrified tubular silica body having desired wall thickness is formed.

Step 5. Rotating and translating, a substrate consisting of porous tubing is heated to a temperature of about 1300 °C and is subjected to plurality of silica particle streams and further reduced dopant particle streams introduced via ports on the chamber. The accelerated particles collide with the substrate and deposit themselves on the substrate. Subsequent particles deposit on the material already deposited and layer by layer the silica member is formed. Porous silica body having about 25-35% solid glass density is obtained by this process.

Step 6. The substrate and/or chamber temperature is raised to about 1400-1600 °C while rotating the substrate and maintained there for certain time interval. A vitrified tubular silica

09881092.061501
105190.2608860

body having desired wall thickness is formed.

Step 7-9. Repeat steps 4-6 further reducing the dopant levels in the deposited silica by lowering the dopant concentrations in the dopant particle streams, etc.

Step 10. The so formed vitrified tubular silica body is heated to temperature of 1300 °C and is subjected to plurality of silica particle stream introduced via ports on the chamber. The so accelerated particles collide with the substrate and deposit themselves on the substrate. Subsequent particles deposit on the material already deposited and layer by layer the silica member is formed. Porous silica body having 25-35% solid glass density is obtained by this process.

Step 11. Introducing silicon tetra fluoride, SiF_4 , through the porous substrate and/or the chamber into the deposited porous silica material for T_1 hours at temperature of about 1100°C - 1400 °C the silica material is doped. T_1 is about 0.3 to 2 hours.

Step 12. The substrate and/or chamber temperature is raised to about 1400-1600 °C while rotating the substrate. The newly deposited porous silica is vitrified, and a tubular silica body having desired doped inner wall thickness IW_t and undoped outer wall OW_t desired wall thickness is formed.

Step 13. The so formed vitrified tubular silica body is heated to a temperature of about 1300 °C and is subjected to plurality of silica particle streams introduced via ports on the chamber. The accelerated particles collide with the substrate and deposit themselves on the substrate. Subsequent particles deposit on the material already deposited and layer by layer the silica member is formed. Porous silica body having about 25-35% solid glass density is obtained by this process

09881092.061501

Step 14. Introducing silicon tetra fluoride, SiF_4 , through the porous substrate and/or the chamber into the deposited porous silica material for $T_2 > T_1$ hours at temperature of about $1100^\circ\text{C} - 1400^\circ\text{C}$ the silica material is doped. T_2 is about 0.4 to 4 hours.

Step 15. The substrate and/or chamber temperature is raised to about $1400-1500^\circ\text{C}$ while rotating the substrate. The newly deposited porous silica is vitrified, and a tubular silica body having desired doped inner wall thickness IW_t and undoped outer wall OW_t desired wall thickness is formed.

Step 16. The so formed vitrified tubular silica body is heated to a temperature of about 1300°C and is subjected to plurality of silica particle streams introduced via ports on the chamber. The accelerated particles collide with the substrate and deposit themselves on the substrate. Subsequent particles deposit on the material already deposited and layer by layer the silica member is formed. A porous silica body having about 25-35% solid glass density is obtained by this process.

Step 17. Introducing silicon tetra fluoride, SiF_4 , through the porous substrate and/or the chamber into the deposited porous silica material for $T_3 > T_2$ hours at temperature of about $1100^\circ\text{C} - 1400^\circ\text{C}$ the silica material is doped. T_3 is about 0.6 to 6 hours.

Step 18. The substrate and/or chamber temperature is raised to about $1400-1600^\circ\text{C}$ while rotating the substrate. The newly deposited porous silica is vitrified, and a tubular silica body having desired doped inner wall thickness IW_t and undoped outer wall OW_t desired wall thickness is formed.

Step 19. The so formed vitrified tubular silica body is heated to a temperature of about 1300°C and is subjected to plurality of silica particle streams introduced via ports on the

chamber. The accelerated particles collide with the substrate and deposit themselves on the substrate. Subsequent particles deposit on the material already deposited and layer by layer the silica member is formed. A porous silica body having 25-35% solid glass density is obtained by this process.

Step 20. Introducing silicon tetra fluoride, SiF_4 , through the porous substrate and/or the chamber into the deposited porous silica material for $T_4 > T_3$ hours at temperature of 1100°C - 1400°C , the silica material is doped. T_4 is about 0.6 to 6 hours

Step 21. The substrate and/or chamber temperature is raised to about 1400 - 1600°C while rotating the substrate. The newly deposited porous silica is vitrified and a tubular silica body having desired doped inner wall thickness IW_t and undoped outer wall OW_t desired wall thickness is formed.

Step 22-24. Repeat Steps 12-14 while further reducing the exposure to gaseous dopant, SiF_4 in this case.

Step 25. The substrate is transferred out of the deposition chamber area and the substrate is removed. If wetting between the substrate and silica occurs, the substrate is heated to the softening point of the silica. The contact between the substrate and the silica member is melted and the substrate is removed.

Step 26. The so formed silica member is collapsed and a solid rod like silica member is formed. Undoped core (high index of refraction material) surrounded by graded index of refraction fluorine doped cladding (low index of refraction material) having desired diameter and length is formed. The duration of the silica deposition for certain substrate cross sections and sizes can be adjusted to allow for various ratios between the core diameter and the outside

09881092-061501
T05190-260T8860

cladding layer diameter of the fiber optic preform, e.g., 1:2, 1:3, 1:5, etc. The length of the chamber and the translation capabilities can provide basis for fabrication fiber optic preforms that are up 6 inches or more in diameter and several meters in length. The radial distribution of the index of refraction in the core and the cladding will depend on the thickness of the doped layer deposited and on the pore density in the as deposited preform.

Example No. 8: Doped core having graded index of refraction and fluorine doped cladding having graded index of refraction fiber optic preform fabrication

Step 1. Rotating and translating, a substrate consisting of porous tubing is heated to a temperature of about 1300 °C and is subjected to plurality of silica and dopant particle streams introduced via ports on the chamber. The accelerated particles collide with the substrate and deposit themselves on the substrate. Subsequent particles deposit on the material already deposited and layer by layer the silica member is formed. A porous silica body having about 25-35% solid glass density is obtained by this process.

Step 2. The substrate and/or chamber temperature is raised to about 1400-1600 °C while rotating the substrate and maintained there for certain time interval. A vitrified tubular silica body having desired wall thickness is formed.

Step 3. Rotating and translating, a substrate consisting of porous tubing is heated to a temperature of about 1300 °C and is subjected to plurality of silica particle stream and reduced concentration dopant particle streams introduced via ports on the chamber. The accelerated

particles collide with the substrate and deposit themselves on the substrate. Subsequent particles deposit on the material already deposited and layer by layer the silica member is formed. A porous silica body having about 25-35% fused silica density is obtained by this process.

Step 4. The substrate and/or chamber temperature is raised to about 1400-1600 °C while rotating the substrate and maintained there for certain time interval. A vitrified tubular silica body having desired wall thickness is formed.

Step 5. Rotating and translating, a substrate consisting of porous tubing is heated to a temperature of about 1300 °C and is subjected to plurality of silica particle streams and further reduced concentration dopant particle stream introduced via ports on the chamber. The so accelerated particles collide with the substrate and deposit themselves on the substrate. Subsequent particles deposit on the material already deposited and layer by layer the silica member is formed. A porous silica body having about 25-35% fused silica density is obtained by this process.

Step 6. The substrate and/or chamber temperature is raised to about 1400-1600 °C while rotating the substrate and maintained there for certain time interval. A vitrified tubular silica body having desired wall thickness is formed.

Step 7-9. Repeat steps 4-6 further reducing the dopant levels in the deposited silica by further lowering the dopant concentrations in the dopant particle stream. Repeat until the desired index of refraction profile in radial direction is obtained.

Step 10. The so formed vitrified tubular silica body is heated to a temperature of about 1380 °C and is subjected to plurality of silica particle streams introduced via ports on the chamber. The accelerated particles collide with the substrate and deposit themselves on the

substrate. Subsequent particles deposit on the material already deposited and layer by layer the silica member is formed. A porous silica body having about 80-90% fused silica density is obtained by this process.

Step 11. The so formed silica body is heated to a temperature of about 1370 °C and is subjected to plurality of silica particle stream introduced via ports on the chamber. The accelerated particles collide with the substrate and deposit themselves on the substrate. Subsequent particles deposit on the material already deposited and layer by layer the silica member is formed. A porous silica body having about 75-85% solid glass density is obtained by this process.

Step 12. The so formed vitrified tubular silica body is heated to temperature of 1360 °C and is subjected to plurality of silica particle stream introduced via ports on the chamber. The so accelerated particles collide with the substrate and deposit themselves on the substrate. Subsequent particles deposit on the material already deposited and layer by layer the silica member is formed. A porous silica body having about 65-75% fused silica density is obtained by this process.

Step 13. The so formed vitrified tubular silica body is heated to a temperature of about 1330 °C and is subjected to plurality of silica particle streams introduced via ports on the chamber. The accelerated particles collide with the substrate and deposit themselves on the substrate. Subsequent particles deposit on the material already deposited and layer by layer the silica member is formed. A porous silica body having about 50-60% fused silica density is obtained by this process.

Step 14. The so formed vitrified tubular silica body is heated to a temperature of about

1300 °C and is subjected to plurality of silica particle streams introduced via ports on the chamber. The accelerated particles collide with the substrate and deposit themselves on the substrate. Subsequent particles deposit on the material already deposited and layer by layer the silica member is formed. A porous silica body having about 25-35% fused silica density is obtained by this process.

Step 15. Introducing silicon tetra fluoride, SiF_4 , through the chamber into the deposited porous silica material for about 0.3 - 6 hours at temperature of 1100°C - 1400°C the silica material is doped. The amount of the SiF_4 penetrating the cladding will be proportional to the pore density and the exposure time at given temperature of the preform.

Step 16. The substrate and/or chamber temperature is raised to about 1400-1600 °C while rotating the substrate. The newly deposited porous silica is vitrified, and a tubular silica body having desired cladding layer wall thickness is formed. Repeat until the desired index of refraction profile in radial direction is obtained.

Step 17. The substrate is transferred out of the deposition chamber area and the substrate is removed. If wetting between the substrate and silica occurs, the substrate is heated to the softening point of the silica. The contact between the substrate and the silica member is melted and the substrate is removed.

Step 18. The so formed silica member is collapsed and a solid rod like silica member is formed. Undoped core (high index of refraction material) surrounded by graded index of refraction fluorine doped cladding (low index of refraction material) having desired diameter and length is formed. The duration of the silica deposition for certain substrate cross sections and sizes can be adjusted to allow for various ratios between the core diameter and the outside

09881092-061501
TOST90-260T8860

cladding layer diameter of the fiber optic preform, e.g., 1:2, 1:3, 1:5, etc. The length of the chamber and the translation capabilities can provide basis for fabrication fiber optic preforms that are up 6 inches or more in diameter and several meters in length. The radial distribution of the index of refraction in the core and the cladding will depend on the thickness of the doped layer deposited and on the pore density in the deposited preform.

***Example No. 9: Fluorine doped cladding having graded index of refraction
fiber optic preform fabrication using prefabricated doped or undoped core rod***

Step 1. Prefabricated silica doped or undoped rod is heated to a temperature of about 1400 °C and is subjected to plurality of silica particle streams introduced via ports on the chamber. The so accelerated particles collide with the substrate and deposit themselves on the substrate. Subsequent particles deposit on the material already deposited and layer by layer the silica member is formed. A porous silica body having about 90-100% fused silica density is obtained by this process.

Step 2. Prefabricated silica doped or undoped rod is heated to a temperature of about 1380 °C and is subjected to plurality of silica particle streams introduced via ports on the chamber. The accelerated particles collide with the substrate and deposit themselves on the substrate. Subsequent particles deposit on the material already deposited and layer by layer the silica member is formed. A porous silica body having about 80-90% fused silica density is obtained by this process.

Step 3. The so formed silica body is heated to a temperature of about 1370 °C and is

subjected to plurality of silica particle streams introduced via ports on the chamber. The accelerated particles collide with the substrate and deposit themselves on the substrate. Subsequent particles deposit on the material already deposited and layer by layer the silica member is formed. A porous silica body having about 75-85% solid glass density is obtained by this process.

Step 4. The so formed vitrified tubular silica body is heated to a temperature of about 1360 °C and is subjected to plurality of silica particle stream introduced via ports on the chamber. The accelerated particles collide with the substrate and deposit themselves on the substrate. Subsequent particles deposit on the material already deposited and layer by layer the silica member is formed. A porous silica body having about 65-75% fused silica density is obtained by this process.

Step 5. The so formed vitrified tubular silica body is heated to a temperature of about 1330 °C and is subjected to plurality of silica particle streams introduced via ports on the chamber. The accelerated particles collide with the substrate and deposit themselves on the substrate. Subsequent particles deposit on the material already deposited and layer by layer the silica member is formed. A porous silica body having about 50-60% fused silica density is obtained by this process.

Step 6. The so formed vitrified tubular silica body is heated to a temperature of about 1300 °C and is subjected to plurality of silica particle streams introduced via ports on the chamber. The accelerated particles collide with the substrate and deposit themselves on the substrate. Subsequent particles deposit on the material already deposited and layer by layer the silica member is formed. A porous silica body having about 25-35% fused silica density is

obtained by this process.

Step 7. Introducing silicon tetra fluoride, SiF_4 , through the chamber into the deposited porous silica material for about 0.3 –6 hours at temperature of about $1100^0 - 1400^0 \text{ } ^\circ\text{C}$ the silica material is doped. The amount of the SiF_4 penetrating the cladding will be proportional to the pore density and the exposure time at given temperature of the preform.

Step 8. The substrate and/or chamber temperature is raised to about $1400\text{-}1600^0 \text{ } ^\circ\text{C}$ while rotating the substrate. The newly deposited porous silica is vitrified, and a tubular silica body having desired cladding layer wall thickness is formed. Repeat until the desired index of refraction profile in radial direction is obtained.

Step 26. The so formed silica member is vitrified and a solid rod like silica member is formed. Doped or undoped core (high index of refraction material) surrounded by graded index of refraction fluorine doped cladding (low index of refraction material) having desired diameter and length is formed. The duration of the silica deposition for certain substrate cross sections and sizes can be adjusted to allow for various ratios between the core diameter and the outside cladding layer diameter of the fiber optic preform, e.g., 1:2, 1:3, 1:5, etc. The length of the chamber and the translation capabilities can provide basis for fabrication fiber optic preforms that are up 6 inches or more in diameter and several meters in length. The radial distribution of the index of refraction in the core and the cladding will depend on the thickness of the doped layer deposited and on the pore density in the as deposited preform.

09881092-061501

Example No. 10: Process for fabrication of fluorine doped cladding tube having graded index of refraction fiber optic preform fabrication

Step 1. Rotating and translating, a substrate consisting of porous tubing is heated to a temperature of about 1400 °C and is subjected to plurality of silica particle streams introduced via ports on the chamber. The accelerated particles collide with the substrate and deposit themselves on the substrate. Subsequent particles deposit on the material already deposited and layer by layer the silica member is formed. A porous silica body having about 90-100% fused silica density is obtained by this process.

Step 2. Prefabricated silica doped or undoped rod is heated to a temperature of about 1380 °C and is subjected to plurality of silica particle streams introduced via ports on the chamber. The accelerated particles collide with the substrate and deposit themselves on the substrate. Subsequent particles deposit on the material already deposited and layer by layer the silica member is formed. A porous silica body having about 80-90% fused silica density is obtained by this process.

Step 3. The so formed silica body is heated to a temperature of about 1370 °C and is subjected to plurality of silica particle streams introduced via ports on the chamber. The so accelerated particles collide with the substrate and deposit themselves on the substrate. Subsequent particles deposit on the material already deposited and layer by layer the silica member is formed. A porous silica body having about 75-85% solid glass density is obtained by this process.

Step 4. The so formed vitrified tubular silica body is heated to a temperature of about 1360 °C and is subjected to plurality of silica particle streams introduced via ports on the

09881092.061501
FOST90.260T8860

chamber. The accelerated particles collide with the substrate and deposit themselves on the substrate. Subsequent particles deposit on the material already deposited and layer by layer the silica member is formed. A porous silica body having about 65-75% fused silica density is obtained by this process.

Step 5. The so formed vitrified tubular silica body is heated to a temperature of about 1330 °C and is subjected to plurality of silica particle streams introduced via ports on the chamber. The accelerated particles collide with the substrate and deposit themselves on the substrate. Subsequent particles deposit on the material already deposited and layer by layer the silica member is formed. A porous silica body having about 50-60% fused silica density is obtained by this process.

Step 6. The so formed vitrified tubular silica body is heated to a temperature of about 1300 °C and is subjected to plurality of silica particle streams introduced via ports on the chamber. The accelerated particles collide with the substrate and deposit themselves on the substrate. Subsequent particles deposit on the material already deposited and layer by layer the silica member is formed. A porous silica body having about 25-35% fused silica density is obtained by this process.

Step 7. Introducing silicon tetra fluoride, SiF_4 , through the porous substrate and the chamber into the deposited porous silica material for about 0.3 - 6 hours at temperature of about 1100°C - 1400 °C, the silica material is doped. The amount of the SiF_4 penetrating the cladding will be proportional to the pore density and the exposure time at given temperature of the preform.

Step 8. The substrate and/or chamber temperature is raised to about 1400-1600 °C while

09881092-061501
T05F90-26018850

rotating the substrate. The porous silica is vitrified and a tubular silica body having desired cladding layer wall thickness is formed.

Step 9. The substrate is transferred out of the deposition chamber area and the substrate is removed. If wetting between the substrate and silica occurs, the substrate is heated to the softening point of the silica. The contact between the substrate and the silica member is melted and the substrate is removed. The duration of the silica deposition for certain substrate cross sections and sizes can be adjusted to allow for various ratios between the inner diameter and the outside diameter of the tubing fiber optic preform, e.g., 1:2, 1:3, 1:5, etc. The length of the chamber and the translation capabilities can provide basis for fabrication doped tubing for fiber optic preforms that are up 12 inches or more in diameter and several meters in length. The radial distribution of the index of refraction in the cladding will depend on the thickness of the doped layer deposited and or the pore density in the as deposited preform.

Example No. 11: Doped core having graded index of refraction for fiber optic preform fabrication

Step 1. Rotating and translating, a substrate consisting of porous tubing is heated to a temperature of about 1300 °C and is subjected to plurality of silica and dopant particle streams introduced via ports on the chamber. The accelerated particles collide with the substrate and deposit themselves on the substrate. Subsequent particles deposit on the material already deposited and layer by layer the silica member is formed. A porous silica body having about 25-35% solid glass density is obtained by this process.

Step 2. The substrate and/or chamber temperature is raised to about 1400-1600 °C while rotating the substrate and maintained there for certain time interval. A vitrified tubular silica body having desired wall thickness is formed.

Step 3. Rotating and translating, a substrate consisting of porous tubing is heated to a temperature of about 1300 °C and is subjected to plurality of silica particle streams and reduced concentration dopant particle stream introduced via ports on the chamber. The so accelerated particles collide with the substrate and deposit themselves on the substrate. Subsequent particles deposit on the material already deposited and layer by layer the silica member is formed. A porous silica body having about 25-35% fused silica density is obtained by this process.

Step 4. The substrate and/or chamber temperature is raised to about 1400-1600 °C while rotating the substrate and maintained there for certain time interval. A vitrified tubular silica body having desired wall thickness is formed.

Step 5. Rotating and translating, a substrate consisting of porous tubing is heated to a temperature of about 1300 °C and is subjected to plurality of silica particle streams and further reduced concentration dopant particle stream introduced via ports on the chamber. The accelerated particles collide with the substrate and deposit themselves on the substrate. Subsequent particles deposit on the material already deposited and layer by layer the silica member is formed. A porous silica body having about 25-35% fused silica density is obtained by this process.

Step 6. The substrate and/or chamber temperature is raised to about 1400-1600 °C while rotating the substrate and maintained there for certain time interval. A vitrified tubular silica body having desired wall thickness is formed.

Step 7-9. Repeat steps 4-6 further reducing the dopant levels in the deposited silica by further lowering the dopant concentrations in the dopant particle stream. Repeat until the desired index of refraction profile in radial direction is obtained.

Step 10. The substrate is transferred out of the deposition chamber area and the substrate is removed. If wetting between the substrate and silica occurs, the substrate is heated to the softening point of the silica. The contact between the substrate and the silica member is melted and the substrate is removed.

Step 11. The so formed silica member is collapsed and a solid rod like silica member is formed. Graded index of refraction core having desired diameter and length is formed. The duration of the silica deposition for certain substrate cross sections and sizes can be adjusted to allow for various ratios between the inner diameter and the outside diameter of the tubing fiber optic preform, e.g., 1:2, 1:3, 1:5, etc. The length of the chamber and the translation capabilities can provide basis for fabrication doped cores for fiber optic preforms that are up 12 inches or more in diameter and several meters in length. The radial distribution of the index of refraction in the cladding will depend on the thickness of the doped layer deposited and on the pore density in the deposited preform.

While the invention has been described with reference to specific embodiments, modifications and variations of the invention may be constructed without departing from the scope of the invention, which is defined in the following claims.